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WEAR RESISTANT COATINGS FOR TITANIUM.(U)

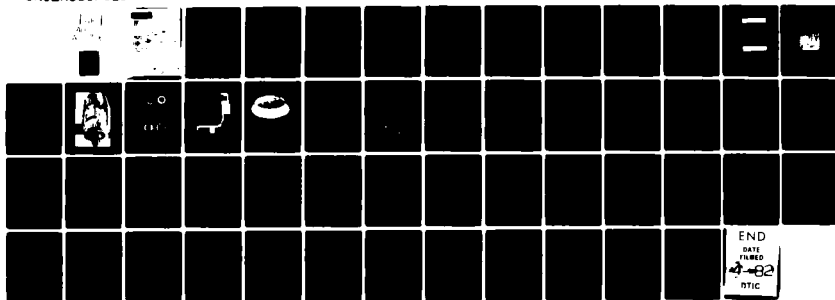
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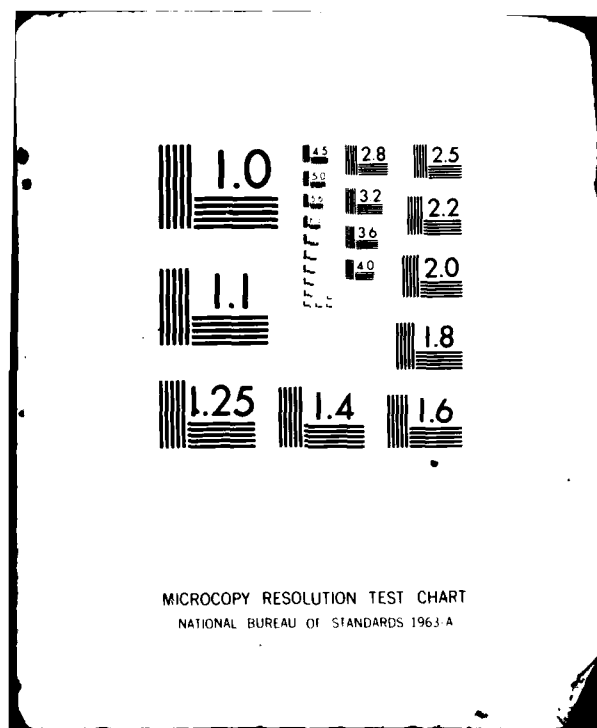
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PREFACE

The authors wish to acknowledge the contributions provided by the following individuals. Messrs. S. Tefft and B. Zelazek conducted the plating and performed wear tests. Mr. T. Brooke assisted with the selection and application of dry-film lubricants. Messrs. G. Lange and G. Hoop were instrumental in arranging engine tests for chromium-molybdenum coated titanium components. Mr. B. Manty provided guidance and advice helpful to the success of this program. Mr. M. Valentine of NAVAIR provided technical monitoring of the program.

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TABLE OF CONTENTS

<i>Section</i>	<i>Page</i>
I INTRODUCTION.....	1
II EXPERIMENTAL PROGRAM.....	3
III CONCLUSIONS.....	24
Evaluation of a New Etching Technique.....	24
Coating of Augmentor Nozzle Support Bearings for Engine Testing.....	24
Evaluation of Various Dry-Film Lubricants.....	24
IV RECOMMENDATIONS.....	25
APPENDIX A — Chromium-Molybdenum Application Procedure for Titanium.....	26
APPENDIX B — Chromium-Molybdenum Application Procedure for Steels	28
APPENDIX C — Suggested Military Specification — Chromium-Molybdenum Plating (Electrodeposited).....	30
ATTACHMENT — Distribution List.....	41
REFERENCES.....	46

LIST OF ILLUSTRATIONS

<i>Figure</i>		<i>Page</i>
1	Bond Strength Test Bar Dimensions.....	4
2	Cross Section of Failure Due to Exceeding of Epoxy Strength.....	5
3	Cross Section of Failure Due to Poor Cr-Mo Diffusion.....	5
4	Apparatus for Plating J52 First-Stage Compressor Blade Roots.....	6
5	P&WA J52 Gas Turbine Engine.....	8
6	Notched Ti 8Al-1Mo-1V Augmentor Nozzle Bearing, Disassembled.....	9
7	Comparison of Notched Titanium and Bill-of-Material Steel Augmentor Nozzle Bearings.....	9
8	Cross Section of No. 1 Bearing Seal Ring Holder.....	10
9	No. 1 Bearing Seal Ring Holder After Cr-Mo Plating and Diffusion Heat Treatment.....	11
10	Schematic Diagram of Falex Lubricant Tester.....	13
11	Exploded View of V-Blocks and Journal Arrangement, Falex Lubricant Tester.....	13

LIST OF TABLES

<i>Table</i>		<i>Page</i>
1	Alloys Used in This Investigation.....	3
2	Falex Wear Tests Using Cr-Mo Coated Ti 6-4 Pins and Uncoated Steel V-Blocks.....	14
3	Solvent Resistance of Dry Film Lubricants, Based on Falex Wear Tests per ASTM-D-2625, Procedure A.....	15
4	Falex Wear Tests Using Cr-Mo Coated Steel Pins and Uncoated Steel V-Blocks.....	16
5	Standards.....	17
6	Instrument Parameters.....	18

SECTION I

INTRODUCTION

This report describes the results of the fourth and final year of a program to develop wear-resistant chromium-molybdenum coatings and to apply these coatings to candidate titanium alloy components. The capability to use titanium for gas turbine or airframe components often depends on a suitable surface treatment being available to alleviate inherent problems such as wear, erosion, and corrosion.

During the first year it was established that a chromium-molybdenum coating can be applied to various titanium alloys as a solution to these problems. Also, because of the compatibility of the solid solutions formed by the chromium-molybdenum-titanium system, the effects of the coating process on the mechanical properties of titanium were minimal. In addition, it was demonstrated that plating with current interruption (pulse plating) can have a marked effect on the coating composition, increasing the molybdenum content.

In the second phase of the program, chromium-molybdenum coatings exhibited vastly superior wear resistance compared to conventional chromium plate. Pulse-plated deposits were found to have even better wear resistance than those produced with direct current on both titanium and steel. Various pulse times were investigated to determine their effect on the coatings. In addition, the chromium-molybdenum coating was found to have no significant effect on the tensile strength, yield strength, or elongation properties of titanium.

Although a reduction in the fatigue strength of one titanium alloy (fully heat-treated Ti 8Al-1Mo-1V) was noted, this was shown to be the result of the heat-treat process. Also, application of the coating increased the creep life up to 50% and protected titanium from oxidation at temperatures up to 760°C (1400°F). Finally, the coating process was successfully scaled up to plate titanium engine sync rings.

During the third program phase, an intensive investigation was undertaken to further evaluate the effect of various pulse settings on the properties of the coatings produced. Variations in cathode efficiency, molybdenum content, surface morphology, and hardness were related to changes in these pulse settings. The effects of plating solution composition and temperature on these properties were also investigated.

Also included was a study of the effect of various pulse settings on the wear resistance of chromium-molybdenum on both titanium and steel. Several heat-treat cycles were evaluated for Ti 8Al-1Mo-1V and Ti 6Al-4V in an effort to maximize adhesion of the coating to the base metal. Further, the corrosion resistance afforded by the coatings was investigated by plating steel roller bearings at various pulse settings and subjecting them to salt-spray testing.

In addition, low-cycle fatigue tests were conducted on chromium-molybdenum coated Ti 8Al-1Mo-1V samples, and fan blade roots were coated as a potential solution to galling problems. As a culmination of the findings to date, a draft of a suggested Military Specification covering chromium-molybdenum coatings for both steel and titanium alloys was prepared.

This fourth phase included the functional testing of two chromium-molybdenum coated engine components (compressor blades and augmentor nozzle support bearings). Also conducted was an intensive evaluation of the influence of various dry-film lubricants when used in conjunction with Cr-Mo. In addition, procedures were established for the application, inspection, and control of the coatings.

The completion of this final phase has:

- Verified the suitability of chromium-molybdenum for use on at least one titanium engine component
- Provided a selection of dry-film lubricants to be used with Cr-Mo for various applications
- Established procedures for the inspection and control of the Cr-Mo plating.

SECTION II

EXPERIMENTAL PROGRAM

The experimental portion of this contract program contains the following:

1. Evaluation of a hot sodium chromate-hydrofluoric acid etch for titanium parts
2. Coating of compressor blade roots with chromium-molybdenum for engine testing
3. Coating of augmentor nozzle support bearings with Cr-Mo for engine endurance test
4. Coating of a bearing seal ring holder with Cr-Mo
5. Evaluation of the wear and solvent resistance afforded by various dry-film lubricants when used in conjunction with Cr-Mo on both titanium and steel
6. Establishment of plating bath analysis procedures
7. Establishment of procedures for inspecting Cr-Mo coatings.

The object of the fourth phase of this program was to optimize the coating process and to test the coating on various engine components.

Materials

Alloys used in this program are listed in Table 1 below:

TABLE 1. ALLOYS USED IN THIS INVESTIGATION

	Type	Test
Titanium Alloys	Ti 6Al-4V	Compressor Blades Seal Ring Holder Wear (Pins)
	Ti 8Al-1Mo-1V	Adhesion Bearings
Steel Alloys	SAE 3135	Wear (Pins)
	AISI 1137	Wear (V-Blocks)

The chemicals used in the preparation of plating solutions and other solutions used in this investigation were all reagent-grade materials, except for the self-regulating chromic acid which was obtained from M&T Chemicals, Inc., under their designation SRHS CR 110.

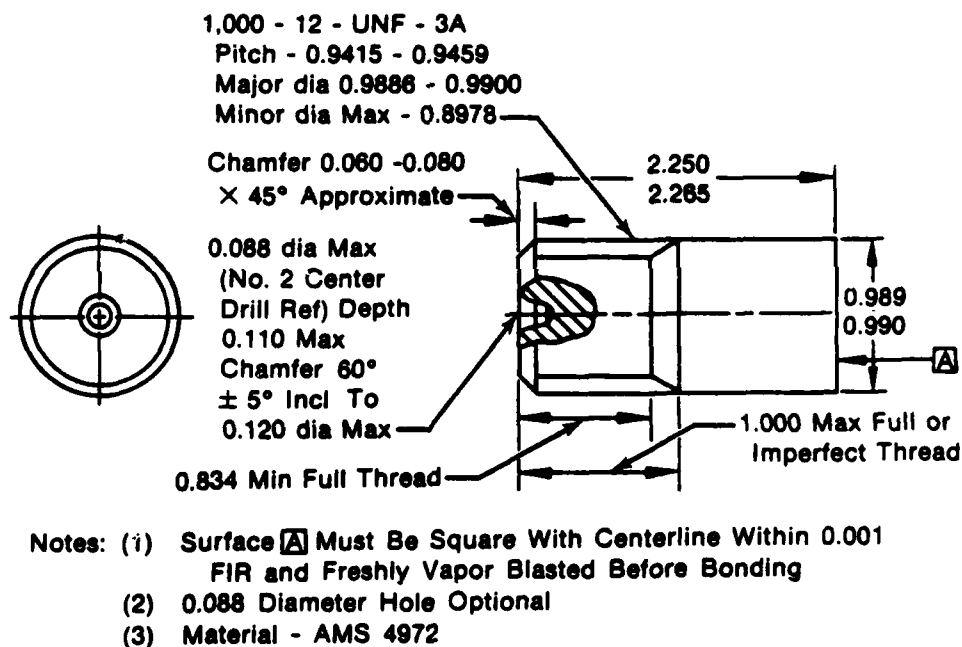
Coating Application Procedure

Titanium compressor blades, bond bars, uniball bearings, wear pins, and seal ring holders were plated and heat-treated in accordance with the procedure detailed in Appendix A. The procedure outlined in Appendix B was employed to coat steel wear pins and uniball bearings.

Evaluation of a New Etching Technique

In the course of preparing the suggested Military Specification for chromium-molybdenum coatings, the concept of using a hot sodium chromate-hydrofluoric acid etch for titanium was encountered (ASTM B481). Although these are the same constituents used previously, the concentrations, temperature, and time of immersion were considerably higher than before. Specifications for this etch are as follows: 250 g/l (33.4 oz/gal) sodium chromate ($\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$), 48 ml/l (6.1 oz/gal) concentrated hydrofluoric acid (48 weight % HF), 82 to 100°C (180 to 212°F), and an immersion time of 20 min. Previously, 20 g/l (2.7 oz/gal) sodium chromate, 2.1 ml/l (0.27 oz/gal) hydrofluoric acid, room temperature, and an immersion time of 15 sec were used. Apparently the increased severity of conditions roughens the surface of the titanium so that it will more readily accept an electroplated coating.

To evaluate the effect of this etch on adhesion, tensile tests of the Cr-Mo/titanium substrate bond were conducted. Bond bars were machined from AMS 4972 (Ti 8Al-1Mo-1V) bar stock per the dimensions shown in Figure 1. The faces (surface A) of six of these bars were hot-etched and then plated with Cr-Mo to a thickness of 0.0013 cm (0.0005 in.). These bars were then heat-treated in a vacuum furnace for 1 hr at 954°C (1750°F) followed by 8 hr at 593°C (1100°F).



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Figure 1. Bond Strength Test Bar Dimensions

Mating bars were epoxy bonded and cured to the coated faces, and these assemblies were subjected to tensile testing. In a previous study, chromium-molybdenum had shown an average yield stress of 42.5 MPa (6160 psi), with a standard deviation of 8.3 MPa (1210 psi). These results were for bars prepared for plating with the conventional etch. For the hot-etched specimens, however, the average yield stress was 74.5 MPa (10,810 psi), with a standard deviation of 18.6 MPa (2700 psi). Furthermore, some of the failures occurred at the epoxy bond between uncoated and Cr-Mo coated bars. This implies that the average bond strength between the chromium-molybdenum electrodeposit and the titanium substrate was even greater. At the least, an increase in bond strength of 75% can be attributed to the use of the hot etch.

Metallographic analysis of the failed specimens showed that three of the six failures occurred because the strength of the epoxy was exceeded. The average yield stress for these specimens was 89.3 MPa (12,950 psi), with a standard deviation of 3.9 MPa (570 psi). Figure 2 shows a cross section of one of these specimens. The chromium-molybdenum layer remains intact and appears uniformly diffused into the titanium substrate.

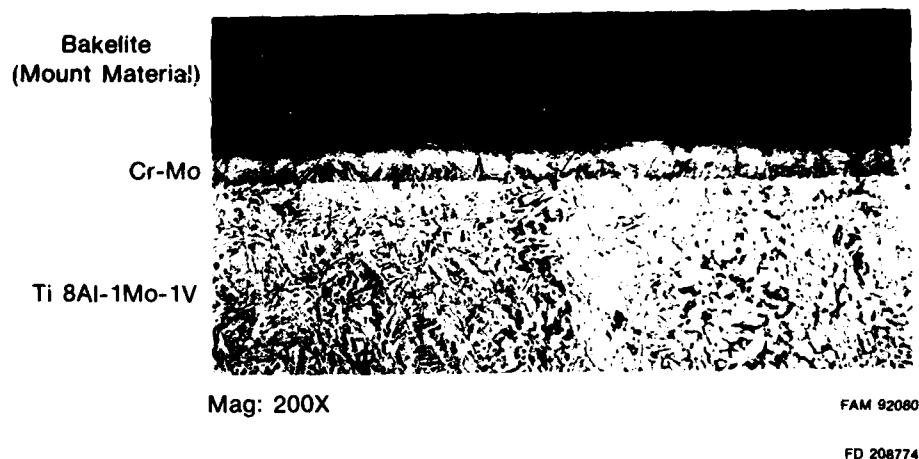


Figure 2. Cross Section of Failure Due to Exceeding of Epoxy Strength

The other three specimens were judged to have failed because of inadequate diffusion. Average yield stress was 59.8 MPa (8670 psi); the standard deviation was 14.0 MPa (2030 psi). Figure 3 shows a typical cross section. It is evident that certain areas of the plating failed to diffuse into the titanium, resulting in large spalls and subsequent coating failure. However, even those specimens exhibiting inadequate diffusion showed a 40% increase in bond strength compared to those etched in the conventional manner.

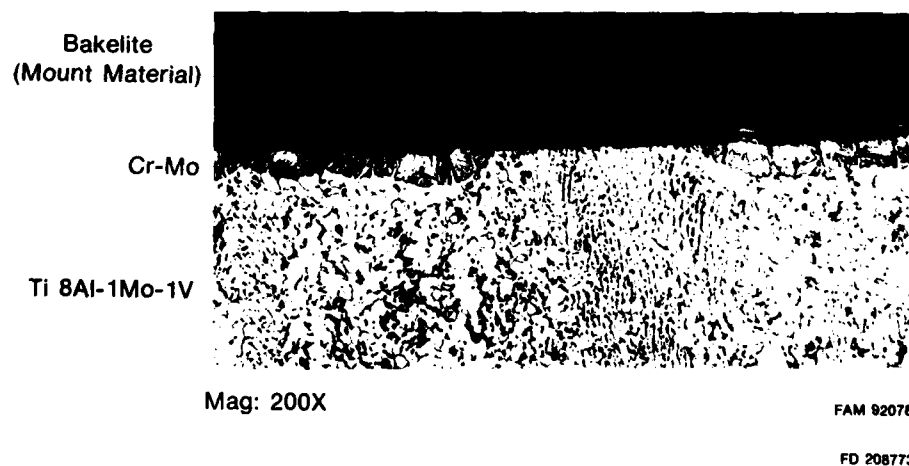


Figure 3. Cross Section of Failure Due to Poor Cr-Mo Diffusion

Coating of Compressor Blade Roots for Engine Testing

For an evaluation of the protection afforded by chromium-molybdenum coatings, a set of 32 1st-stage compressor blades from a Pratt & Whitney J52 engine was obtained. These blades are made of Ti 6Al-4V and have experienced galling problems in the past. The root areas of these blades are normally coated with 0.0025 cm (0.001 in.) of PWA 474 dry-film lubricant.⁽¹⁾ Eleven blade roots were left in this Bill-of-Material condition. Eleven others were plated with another wear-resistant coating, electroless nickel-boron, to a thickness of 0.0013 cm (0.0005 in.) and then coated with 0.0013 cm of PWA 474. The remaining 10 blade roots were electroplated with 0.0013 cm of chromium-molybdenum and then coated with an additional 0.0013 cm of PWA 474. The apparatus used for plating the roots with Cr-Mo is shown in Figure 4.

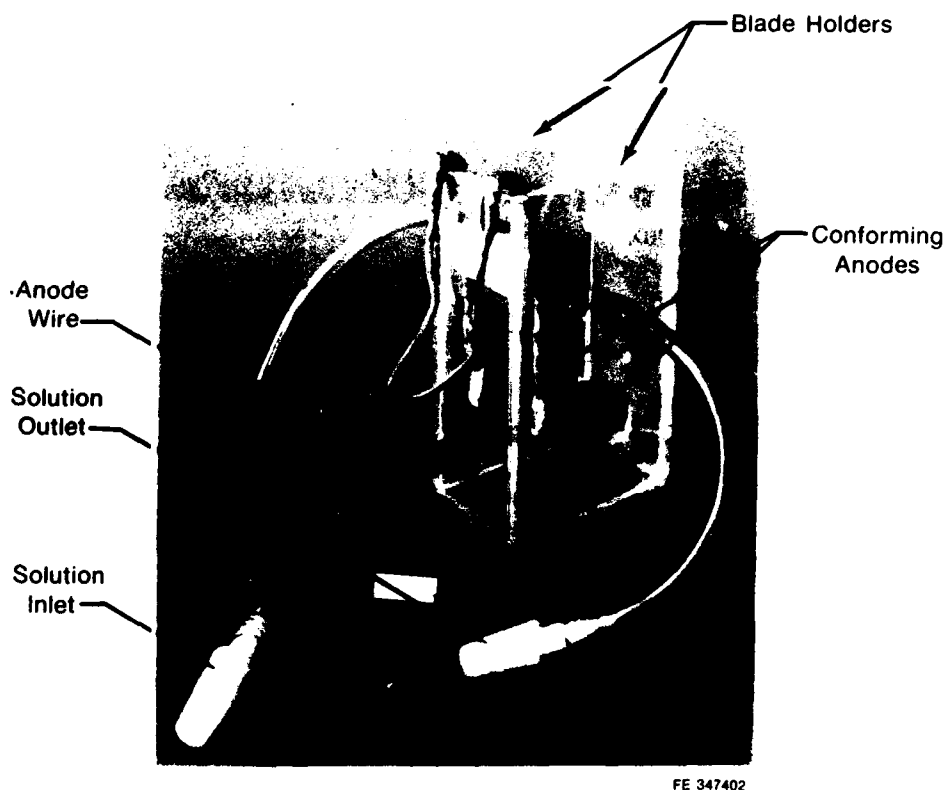


Figure 4. Apparatus for Plating J52 First-Stage Compressor Blade Roots

This apparatus was constructed as follows: A blade root was dipped into molten lead, which was then allowed to solidify. The lead was carefully cut from the blade so that two conforming mirror-image anodes were produced. These were then filed down to provide optimum throwing power into the recessed areas of the blade root. A plexiglass box was constructed to hold the anodes and blade firmly in place. The blades were held by the tips of their midspan shrouds to ensure consistent positioning of the roots between the two anodes. A pump was connected to the apparatus to keep the plating solution well agitated. The roots were plated and heat-treated using the procedure outlined in Appendix A.

⁽¹⁾Dow Corning Corporation, 3400 A

After application of PWA 474, the blades were assembled into engine P-652 for a 168-hr endurance test. A cutaway view of a J52 engine is shown in Figure 5. The engine test was originally scheduled for the spring of 1980, but a lack of funding resulted in its indefinite postponement. The engine was, however, subjected to a 3-hr instrumentation run with no problems.

Coating of Augmentor Nozzle Support Bearings for Engine Testing

Self-aligning bearings, commonly referred to as uniballs, are utilized in numerous gas turbine engine applications. Of particular interest are those used on the augmentor nozzle supports of the Pratt & Whitney F100 engine. The convergent flaps of the augmentor nozzle are held in place by these bearings. Although they are fabricated from stainless steel alloys, these bearings remain susceptible to corrosion because of the hot environment to which they are exposed. The feasibility of using titanium uniballs for this application is therefore being evaluated. To combat the inherent tendency of titanium to gall when worn against itself, bearing surfaces are coated with chromium-molybdenum.

To allow the outside diameter of the ball and/or the inside diameter of the bearing race to be plated, a notched race was designed. This allows the ball to be removed easily. A number of these uniballs were fabricated from AMS 4972 (Ti 8Al-1Mo-1V) bar stock. Figure 6 illustrates one of these bearings. To provide room for the notches, the size of the titanium ball was slightly reduced while the wall thickness of the race was increased. This can be seen in Figure 7, which compares a notched titanium uniball to a Bill-of-Material steel uniball. The steel races are swaged onto the balls and cannot be removed.

Ten of the titanium balls were plated on the outside diameter to a thickness of 0.0013 cm (0.0005 in.) with Cr-Mo per Appendix A. PWA 550⁽¹⁾ dry-film lubricant was then applied, and the balls were replaced in their races. These uniball assemblies were subjected to a 520-hr endurance test in an F100 engine, P-265X-13. After the test, seven of twenty steel balls had "frozen" to their races, but none of the ten Cr-Mo coated titanium bearings had failed. Because of this excellent performance, chromium-molybdenum coatings will continue to be evaluated for this application, both on a test rig and on experimental engines.

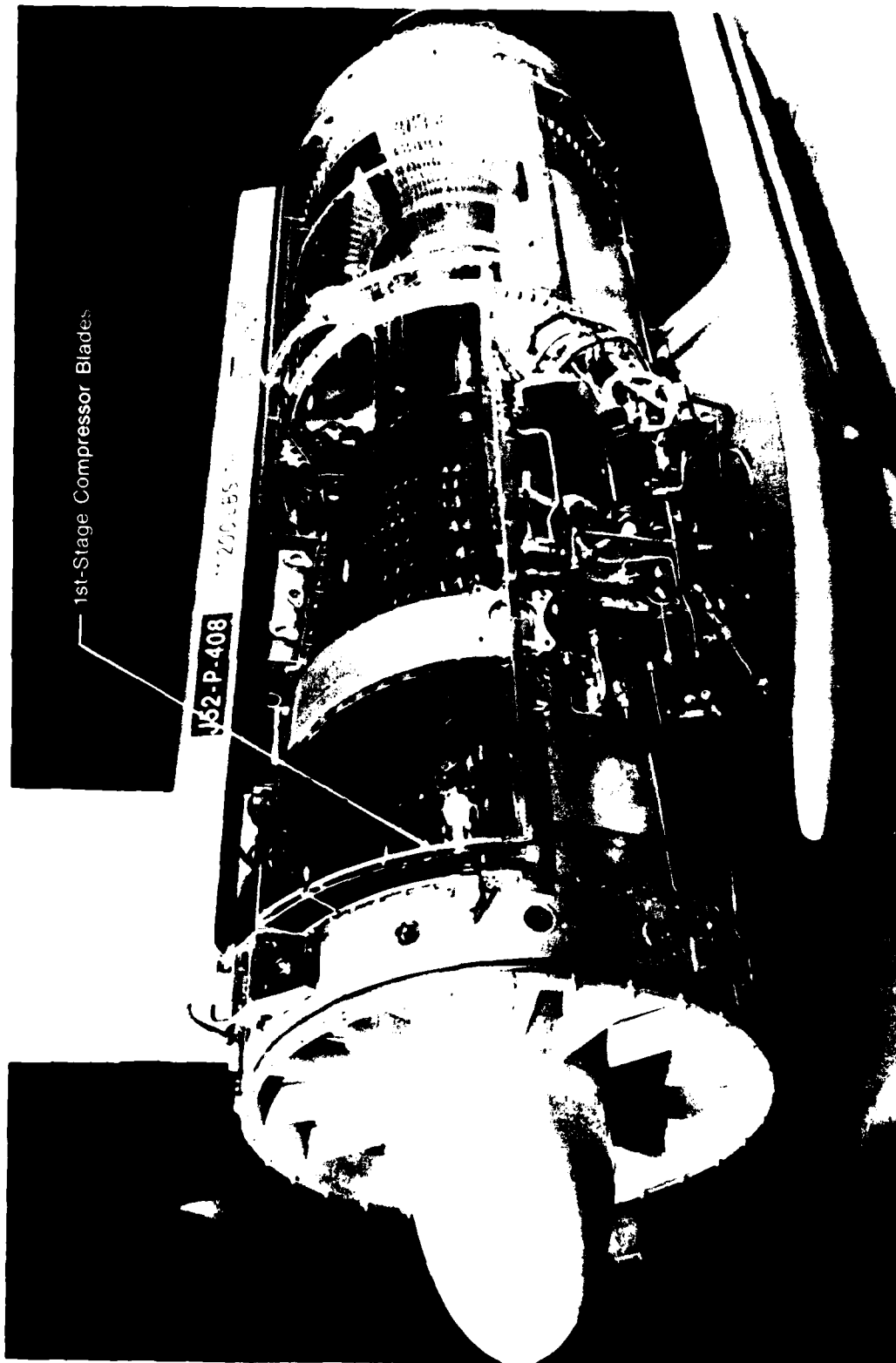
Coating of Bearing Seal Ring Holder

Another engine component on which the use of a chromium-molybdenum coating is being considered is the No. 1 bearing seal ring holder of the F100 engine. This component is made of Ti 6Al-4V and has experienced the problem of the seal ring wearing into the contact surface. This problem area is shown in Figure 8, a cross-sectional view of the component. The Project Engineering Department proposed application of a three-layer coating to build up these worn surfaces during engine overhaul: 0.0013 to 0.0038 cm chromium-molybdenum, then 0.025 to 0.038 cm electrolytic nickel (AMS 2424), and finally, 0.0025 to 0.0038 cm electroless nickel (AMS 2404). The Cr-Mo layer was specified because the plating process has been proven effective in preventing hydrogen embrittlement.

To plate the worn surface with Cr-Mo, the entire part was first coated with a rubberlike masking material.⁽²⁾ Lead wire was then secured around the perimeter of the part as an anode to provide throwing power into the area to be plated. The masking was carefully peeled from the worn area, which was then vapor blasted and etched. The entire part was immersed into the Cr-Mo bath and plated per Appendix A. The masking was then removed, and the part was diffusion heat-treated in a vacuum furnace for 3 hr at 760°C (1400°F) followed by 6 hr at 538°C (1000°F). Figure 9 shows the seal ring holder after the completion of this process.

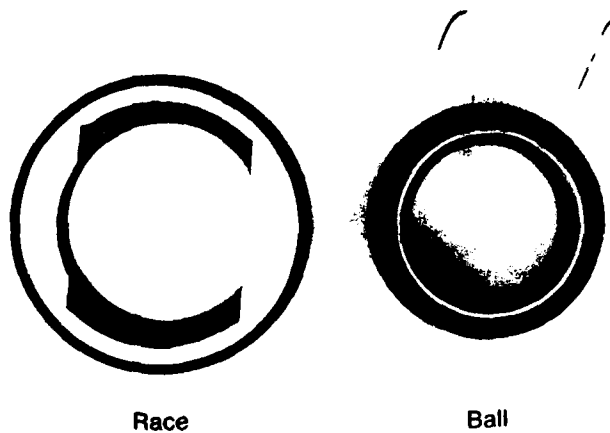
⁽¹⁾Electrofilm Incorporated, Lubri-Bond HT

⁽²⁾Turco Products, Turcoform MASK-522.



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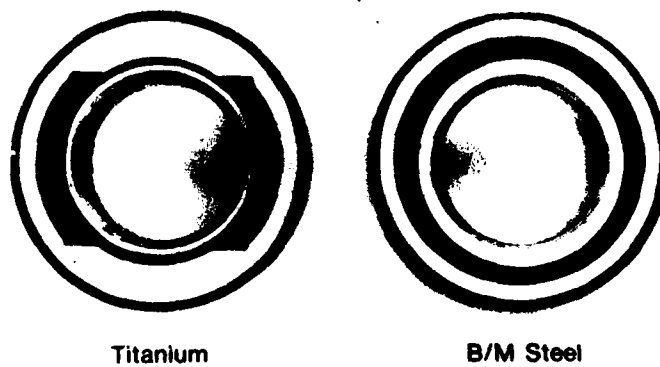
Figure 5. P&WA J52 Gas Turbine Engine



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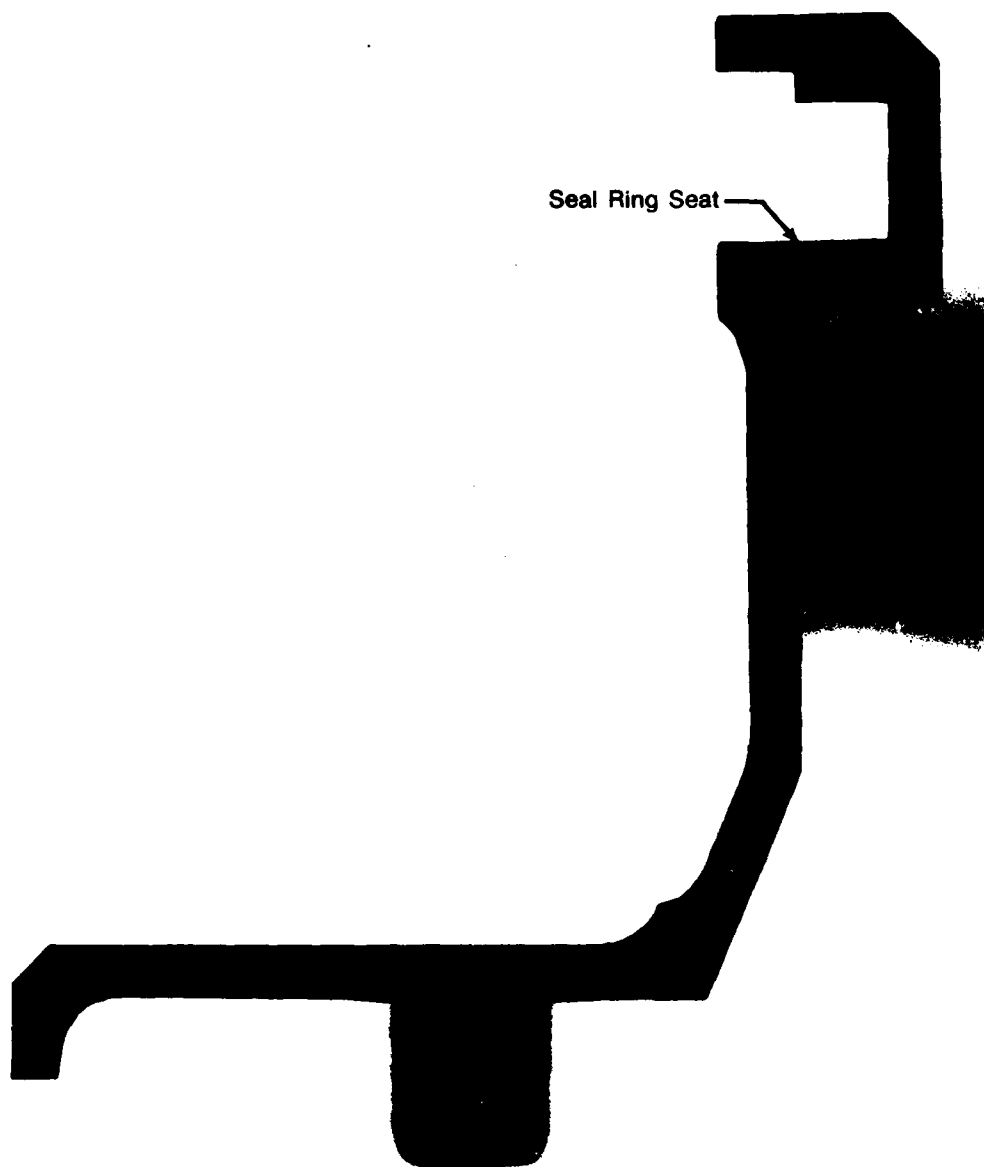
Figure 6. Notched Ti 8Al-1Mo-1V Augmentor Nozzle Bearing, Disassembled



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Figure 7. Comparison of Notched Titanium and Bill-of-Material Steel Augmentor Nozzle Bearings



Mag: 4½X

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Figure 8. Cross Section of No. 1 Bearing Seal Ring Holder



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Figure 9. No. 1 Bearing Seal Ring Holder After Cr-Mo Plating and Diffusion Heat Treatment

Unfortunately, the plan to use the three-layer coating was abandoned due to the many processing steps involved. However, the job demonstrated the capability to Cr-Mo coat titanium parts of complex geometry. More importantly, it suggests the possibility of coating new seal ring holders with chromium-molybdenum to prevent the wear problem.

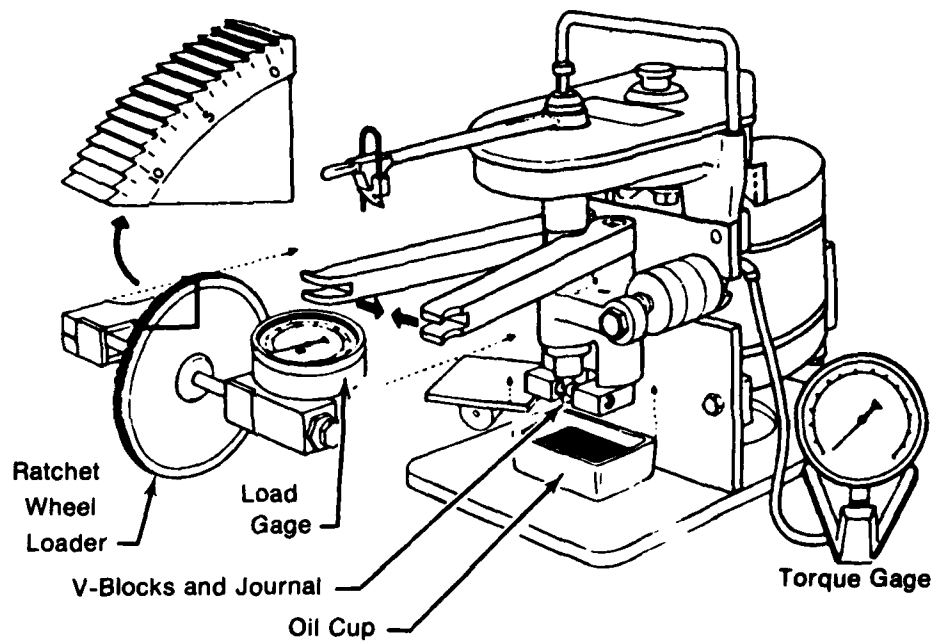
Evaluation of Various Dry-Film Lubricants

To ascertain the effects of various dry-film lubricants on the wear resistance of chromium-molybdenum, 30 Ti 6Al-4V Falex test pins were pulse-plated at 3 ms "on"/3 ms "off" with 0.0013 cm (0.0005 in.) of Cr-Mo. Ten lubricants were selected, six of which are covered by Pratt & Whitney Aircraft specifications:

- PWA 474 --- Dow Corning Corporation, 3400A
- PWA 550 --- Electrofilm Incorporated, Lubri-Bond HT
- PWA 585 — (a) Molybdenum disulfide powder — Dow Corning Corporation, Molykote Z
(b) Graphite powder — Joseph Dixon Crucible Company, Microfyne Flake Graphite
(c) Sodium silicate solution, 47°Be' — PQ Corporation, "K" Sodium Silicate
- PWA 586 — Fel-Pro Incorporated, C-200
- PWA 36035 — Fel-Pro Incorporated, C-300
- PWA 36070 — Electrofilm Incorporated, Lube-Lok 2006
- Lubeco Incorporated, K-350
- Lubeco Incorporated, M-390
- E/M Lubricants Incorporated, Esnalube 382
- E/M Lubricants Incorporated, Ecoalube 642.

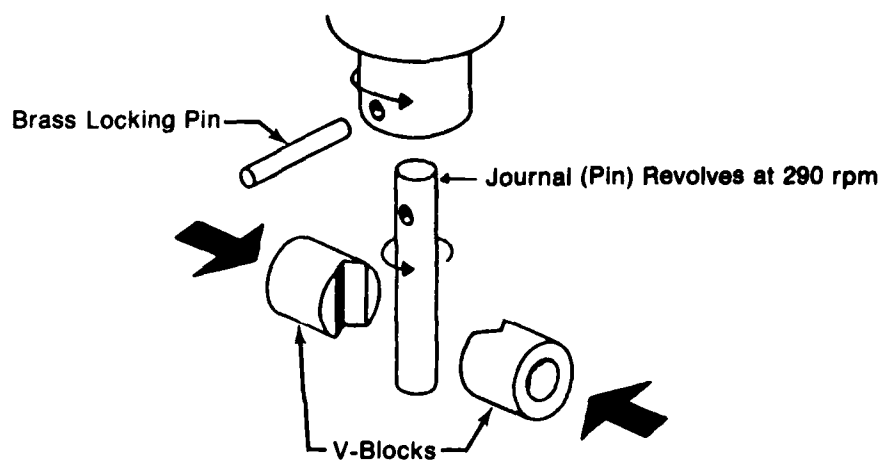
Each of these lubricants was applied to three Cr-Mo plated pins. These pins were tested with a Falex wear tester. This equipment is universally accepted as a method for screening wear couples. While results are not always correlatable to in-service conditions, it does provide a somewhat controlled method of materials comparison by providing fixed specimen geometry.

The Falex wear testing machine is shown in Figures 10 and 11. A 0.635-cm dia journal (pin) is rotated against two stationary V-blocks to give a four-line contact. The test pieces and their supporting jaws are immersed in the oil sample cup or run dry. Coatings or dry-film lubricants to be investigated are applied to the pins and/or V-blocks. The pin is rotated at 290 rpm and a load is applied to the V-blocks through a nutcracker action lever arm and spring gage. The load is applied by means of a ratchet wheel mechanism that also may be used to indicate the extent of wear like a micrometer. An 18-tooth advance on the ratchet wheel is equal to 0.0025 cm of wear. The entire load arm assembly is free to rotate about the main shaft, and friction developed during the test is shown in inch-pounds on the torque gage.



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Figure 10. Schematic Diagram of Falex Lubricant Tester



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Figure 11. Exploded View of V-Blocks and Journal Arrangement, Falex Lubricant Tester

In the test method selected, the V-block load was increased incrementally according to the schedule given in ASTM-D-2625 Procedure A. It consists of running two stationary V-block specimens against a rotating pin until a sharp increase of 1.13 N-m (10 in.-lb) in steady-state torque or pin breakage is experienced. A prescribed schedule of jaw load application and dwell time on the rotating pin is followed: 3 min at 1330 N plus 1 min at 2220 N plus 1 min at 3330 N plus 4450 N until failure.

For this study, the pins were run dry against uncoated AISI 1137 steel V-blocks. Results of these tests are shown in Table 2. These results show that PWA 36035 (Fel-Pro C-300) provides excellent wear resistance when used in conjunction with chromium-molybdenum. This dry-film lubricant is resin-bonded and is alleged to provide wear resistance at temperatures from -55 to 650°C (-65 to 1200°F). It can be air dried, but works best when cured at 260°C (500°F) for 30 min. Another air-dried lubricant, PWA 550 (Lubri-Bond HT), also fared well in the test. The primary drawback to these two air-dried lubricants is that they offer little solvent resistance, making them susceptible to removal by fuels, cleaning fluids, etc. Heat-cured, resin-bonded, dry-film lubricants exhibit superior solvent resistance compared to those that are air-dried (Reference 1). Among these, PWA 474 (Dow Corning 3400A), Ecoalube 642, and Lubeco K-350 showed the best results when used with Cr-Mo.

The two inorganic-bonded lubricants evaluated in these tests (PWA 585 and Esnalube 382) deserve some mention. The pins coated with these lubricants did not fail in the usual manner (a sudden increase in torque of over 1.13 N-m). Instead, the torque reading steadily climbed until a very high torque of 9.0 N-m was reached. By this time the pin and V-blocks were so hot that the wear tester had to be shut off. This high torque and heating up of the specimens shows that there is a considerably higher coefficient of friction for inorganic-bonded lubricants than for those that are resin-bonded. However, the pins did not appear to be galled, unlike conventional failures. This shows that the lubricants provided high-temperature protection. Indeed, inorganic-bonded lubricants are noted for their high-temperature resistance, and also for their high load resistance (Reference 1). They may be suitable for use in applications where movement of coated parts is minimal.

TABLE 2. FALEX WEAR TESTS USING Cr-Mo COATED Ti-6-4 PINS AND UNCOATED STEEL V-BLOCKS

Dry-Film Lubricant	Average Minutes to Failure at Specified Loads				Standard Deviation
	1330N (300 lb _f)	2220N (500 lb _f)	3330N (750 lb _f)	4450N (1000 lb _f)	
PWA 474	—	—	—	54	42
PWA 550	—	—	—	63	12
PWA 585	—	—	—	9	0.4
PWA 586	—	—	—	3	*
PWA 36035	—	—	—	227	29
PWA 36070	1	—	—	—	*
Lubeco K-350	—	—	—	48	25
Lubeco M-390	—	0.2	—	—	*
Esnalube 382	—	—	—	6	0.9
Ecoalube 642	—	—	—	74	76

*Standard deviations for these tests are misleading because each set included at least one pin which failed to reach 4450N.

On the basis of the Falex wear results, as shown in Table 2, five lubricants were selected for further evaluation. These included PWA 474, PWA 550, PWA 36035, Lubeco K-350, and Ecoalube 642. Since engine parts can come into contact with a variety of solvents, the resistance of each lubricant to the following was ascertained:

- Fuel: MIL-T-5624, Type JP-4
- Oil: MIL-L-7808
- Alkaline Rust Remover: 120-360 g/l Kelite No. 235, The Richardson Company, Allied-Kelite Products Division
- Gas Path Cleaner: 20% B&B 3100, B&B Chemical Company, Incorporated
- Sea Water: 3.194% NaCl, I.A.P.O. Standard Sea-Water Service.

Ti 6Al-4V test pins were plated with chromium-molybdenum, heat-treated, lightly vapor blasted, and then coated with the various dry-film lubricants as before. Falex wear tests were repeated; but, before testing, each pin was immersed for 5 min in one of the solvents. Then the results were compared to the data from Table 2, and a subjective rating of the resistance of each lubricant to each solvent was made. Table 3 illustrates these results. PWA 474 proved to be far superior to the other lubricants, but Ecoalube 642 and possibly Lubeco K-350 may be acceptable in some cases. All three of these are heat-cured, resin-bonded lubricants. PWA 36035, which performed so well in the first phase of testing, is apparently useful only in dry environments. PWA 550, although resistant to alkalies and salt water, is soluble in organics. The advantages of PWA 36035 and 550 are that they do not have to be heat-cured, making them ideal for temperature-sensitive parts.

To identify the dry-film lubricant most suitable for use with Cr-Mo coated steel, 15 SAE 3135 steel pins were pulse-plated at 59.9 ms "on"/99.9 ms "off" to a thickness of 0.0013 cm (0.0005 in.) per the procedure outlined in Appendix B. The five lubricants evaluated for solvent resistance were again applied, and Falex wear tests were run using AISI 1137 steel V-blocks. Results are summarized in Table 4. Once again, PWA 36035 (Fel-Pro C-300) provided the best wear resistance, but results were not as good as those for titanium pins. PWA 474 (Dow Corning 3400A), however, showed virtually the same results as before (52 min at a load of 4450N for steel pins vs 54 min at this load for titanium pins). In view of the excellent solvent resistance afforded by this lubricant, it is recommended for use with chromium-molybdenum on both titanium and steel alloy substrates.

TABLE 3. SOLVENT RESISTANCE OF DRY FILM LUBRICANTS, BASED ON FALEX WEAR TESTS PER ASTM-D-2625, PROCEDURE A

Dry-Film Lubricant	Solvent				
	JP-4 Fuel	7808 Oil	Kelite 235	B&B 3100	Sea Water
PWA 474	Excellent	Excellent	Excellent	Good	Good
Ecoalube 642	Good	Fair	Fair	Excellent	Excellent
Lubeco K-350	Excellent	Good	Excellent	Poor	Poor
PWA 550	Poor	None	Excellent	None	Excellent
PWA 36035	Poor	Poor	None	Fair	Poor

TABLE 4. FALEX WEAR TESTS USING Cr-Mo COATED STEEL PINS AND UNCOATED STEEL V-BLOCKS

Dry-Film Lubricant	Average Minutes to Failure at Specified Loads				Standard Deviation
	1330N (300 lb _f)	2220N (500 lb _f)	3330N (750 lb _f)	4450N (1000 lb _f)	
PWA 474	—	—	—	52	18
PWA 550	—	—	—	2	•
PWA 36035	—	—	—	92	73
Lubeco K-350	—	—	—	12	14
Ecoalube 642	—	—	—	15	3

*Standard deviation for this test is misleading because some pins failed to reach 4450N.

Plating Bath Analysis Procedure

In order to monitor the condition of the chromium-molybdenum plating bath, a method of analysis is needed. To maintain flexibility, both atomic absorption and wet chemistry methods are provided.

1. Atomic Absorption Method: Scope

This method provides for the analysis of chromium (Cr), molybdenum (Mo), and sulfate (SO₄) in the plating solution described below.

- a. Composition:

Unichrome ⁽¹⁾	300 g/l (40 oz./gal)
Ammonium Molybdate (NH ₄) ₆ Mo ₇ O ₂₄ · 4H ₂ O	75 g/l (10 oz./gal)
- b. Operating Control Range:

Chromium	121 g/l to 148 g/l
Molybdenum	Nominal: 40.8 g/l
Sulfate	1.05 to 2.10 g/l

2. Apparatus

Atomic Absorption Spectrophotometer (AA), Perkin-Elmer Model 603 or equivalent.

3. Reagents

High purity metals as necessary for nominal composition, Spex or equivalent, or certified aqueous 1000 ppm standards, Fisher or equivalent.

Barium chloride solution. Prepare by dissolving 0.1779g of barium chloride, BaCl₂ · 2H₂O, in deionized water and diluting to 500 ml. This solution contains 200 ppm Ba.

Hydrochloric acid, HCl, concentrated.

Potassium chloride solution. Prepare by dissolving 5.0g of potassium chloride, KCl, in 100 ml of deionized water.

⁽¹⁾M&T Chemicals, Inc., SRHS CR 110.

4. Procedure

a. Standard Preparation

1. Using 1000 ppm stock standards, pipet 10.0 ml each of Cr and Mo into separate 100 ml glass volumetric flasks. Fill to the mark with deionized water, cap, and mix well. These solutions now contain 100 ppm Cr and Mo, respectively.
2. Dissolve 1.479g of anhydrous sodium sulfate, Na_2SO_4 , in deionized water and dilute to one liter. This solution contains 1000 ppm sulfate, SO_4 .
3. Working standards are prepared as shown in Table 5 below. From the Cr, Mo, and SO_4 solutions prepared above, accurately pipet a series of four standards and transfer to 100 ml volumetric flasks. Dilute to volume with deionized water and mix well.

b. Test Specimen Preparation

1. *Chromium analysis sample.* Pipet 1.0 ml of sample into a 1000 ml volumetric flask. Dilute to mark with deionized water and mix well.
2. From this dilution, pipet 1.0 ml into a 50 ml volumetric flask, add 1.0g NH_4Cl , and dilute to mark with deionized water. Mix well.
3. *Molybdenum analysis sample.* Pipet 0.5 ml of sample into a 1000 ml volumetric flask. Dilute to mark with deionized water and mix well.
4. Take a 100 ml aliquot from this dilution, add 2.0g NH_4Cl , and mix well.
5. *Sulfate analysis sample.* Pipet 25 ml of sample into a 1000 ml volumetric flask. Dilute to mark with deionized water and mix well.
6. Pipet 10 ml of this dilution into a 25 ml volumetric flask. Add 0.5g NH_4Cl , one drop of concentrated HCl , one ml of KCl solution, and 10.0 ml of the 200 ppm Ba solution. Make to volume, shake vigorously for 2 to 3 min and allow to stand overnight. Treat, in exactly the same way, 10-ml aliquots of the working standards and a blank (deionized water). After standing, carefully decant about 10 ml of the supernatant into a clean flask or beaker.
7. It is extremely important to make all dilutions as required. Should any deviation be necessary, standard values will have to be corrected accordingly.

TABLE 5. STANDARDS

Standard	Cr (ml)	Cr conc., (mg/l)	Mo (ml)	Mo conc., (mg/l)	SO_4 (ml)	SO_4 conc., (mg/l)	$\text{NH}_4\text{Cl}^{(5)}$ (g)
1 (Blank)	0.0	0.0	0.0	0.0	0.0	0.0	2.0
2	1.0	1.0	10.0	10.0	1.0	10.0	2.0
3	2.0	2.0	20.0	20.0	2.0	20.0	2.0
4	4.0	4.0	40.0	40.0	4.0	40.0	2.0

⁽⁵⁾ NH_4Cl is a matrix modifier to improve precision and accuracy of analysis. Nominal concentration should be kept at 2.0%.

c. Instrumental Conditions and Readings

1. Set the instrument parameters, as shown in Table 6 below, and make the necessary adjustments to maximize absorbance. Refer to instrument manufacturer's operator's guide if necessary.
2. Aspirate the standards and appropriate test specimens; record the readings.
3. Plot a calibration graph of absorbance vs concentration for each constituent. From the resulting curve, read the concentrations of the diluted samples.
4. Obtain the concentration of each constituent in the plating bath as follows:

Chromium — multiply Cr concentration of diluted sample by 50,000
Molybdenum — multiply Mo concentration of diluted sample by 2000
Sulfate — multiply Ba concentration of diluted sample by 100.

TABLE 6. INSTRUMENT PARAMETERS

	<i>Cr</i>	<i>Mo</i>	<i>Ba (SO₄)</i>
Wavelength (nm)	357.9	313.0	553.6
Slit setting (nm)	0.7(4)	0.7(4)	0.14(2)
Wavelength range	UV	UV	UV
Operation mode ⁽⁶⁾	Absorbance	Absorbance	Absorbance
Flame gases	C ₂ H ₂ -N ₂ O	C ₂ H ₂ -N ₂ O	C ₂ H ₂ -N ₂ O
Burner head	N ₂ O	N ₂ O	N ₂ O
Burner head orientation	Normal	Normal	Normal

⁽⁶⁾In the event automatic concentration readout is available, the mode would be concentration. It is desirable to calibrate in this mode.

1. Wet Chemistry Method: Scope

This method provides for the analysis of chromium (Cr), molybdenum (Mo), and sulfate (SO₄) in the plating solution described below.

- a. Composition:
Unichrome⁽⁷⁾ 300 g/l (40 oz./gal)
Ammonium Molybdate 75 g/l (10 oz./gal)
(NH₄)₆ Mo₇ O₂₄ · 4H₂O
- b. Operating Control Range:
Chromium 121 g/l to 148 g/l
Molybdenum Nominal: 40.8 g/l
Sulfate 1.05 to 2.10 g/l

2. Apparatus

Centrifuge, Ion-exchange column.

⁽⁷⁾M&T Chemicals, Inc., SRHS CR 110.

3. Solutions

- a. *Starch solution:* Combine 1.0g starch and a small amount of water. Add 150 ml hot water and boil for 2 to 3 minutes.
- b. *Sodium thiosulfate (0.1N):* Prepare a 1 liter solution of 25g $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ in water. Add 0.1g Na_2CO_3 and allow to stand for one week.

● **Standardization:**

1. Primary standard potassium dichromate 0.1N
 - a. Dry sample of potassium dichromate (NBS 136) 0.1 N at 300 to 350°F for 1 to 1½ hours. Weigh out 4.9040g precisely.
 - b. Thoroughly mix potassium dichromate in water to make 1 liter.
2. Standardization
 - a. Pipette three 25 ml samples of primary potassium dichromate solution into three 500 ml glass-stoppered flasks.
 - b. Add 100 ml water, 2 to 3g potassium iodide, and 10 ml HCl.
 - c. Mix thoroughly in stoppered flask and let stand in a closed cabinet for 8 to 10 minutes.
 - d. Add 150 to 200 ml H_2O , rinsing flask sides.
 - e. Titrate with sodium thiosulfate, swirling continuously, until color change from reddish brown to yellow green occurs.
 - f. Add 4 to 6 ml starch solution and titrate accurately until color changes from blue to light green.
 - g. Record titers and average.
 - h. Calculations:
$$\begin{aligned} N &= \text{Normality of sodium thiosulfate} \\ V &= \text{Volume of sodium thiosulfate} \\ W &= \text{Normality of potassium dichromate} \\ P &= \text{Volume of potassium dichromate} \\ N &= \frac{WP}{V} \end{aligned}$$

- c. *Barium chloride (10%)*: Dissolve 100g BaCl_2 in water to make 1 liter.
- d. *Sulfate solution "A"*: Prepare a 1 liter solution with water and 420 ml HCl.
- e. *Sulfate solution "B"*: Prepare a 1 liter solution with water and 300g $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$.
- f. *Potassium iodide solution (10%)*: Prepare a 1 liter solution with water and 100g KI. Add 1.0g KOH and store in dark bottle.
- g. *Ammonium bifluoride solution (10%)*: Prepare a 1 liter solution with water and 100g NH_4HF_2 . Store in plastic container.
- h. *Buffer solution, pH5, (0.5 M $\text{HC}_2\text{H}_3\text{O}_2$ - 0.5 M $\text{NaC}_2\text{H}_3\text{O}_2$)*: Prepare a 1 liter solution with distilled water, 29 ml glacial acetic acid, and 68g $\text{NaC}_2\text{H}_3\text{O}_2 \cdot 3\text{H}_2\text{O}$.

4. Procedure

a. Chromic Acid

1. Dilute a 10 ml test specimen with water in a 250 ml volumetric flask.
2. Pipette 10 ml of this diluted solution into a 250 ml beaker.
3. Add 10 ml NH_4HF_2 solution, 50 to 100 ml water, and 25 ml KI solution.
4. Add 10 ml H_2SO_4 (1:1) and immediately titrate with standard $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ solution to light yellow-green color.
5. Add 1 to 2 ml of starch solution, observing sharp color change from dark blue to clear green. Record titer.
6. Calculations:

$$\text{g/l chromic acid (CrO}_3\text{)} = ab (1.48)$$

where

a = volume sodium thiosulfate (ml)

b = normality sodium thiosulfate

$$\text{g/l hexavalent chromium (Cr}^{6+}\text{)} = \text{g/l CrO}_3 \times 0.52$$

b. Molybdenum⁽¹⁾

1. *Column preparation.* The ion-exchange column consists of 40.6 cm lengths of 2.54 cm ID polystyrene tubing with a 0.64 cm wall. The lower end of the column is closed off with a section of Lucite rod with a 0.48 cm hole drilled through its axis. A section 2.54 cm long on the end of the Lucite rod is machined down to a 0.95 cm diameter so as to permit the connection of a short length of polyethylene tubing to the column. The other end of the Lucite rod, which is glued to the bottom of the column, is machined so as to have a 45 deg taper from the 2.54 cm ID of the column to the 0.48 cm diameter hole through the rod. This taper eliminates the holdup of the eluted species in the "corner" at the bottom of the column. A short length of 0.64 cm polystyrene tubing is inserted into the wall of the column about 5 cm from the top. This piece of tubing allows the connection of the column to an overhead gravity feed by means of polyethylene tubing. The top of the column is sealed with a rubber stopper when the overhead gravity feed is in use. The top of the column should also be threaded and capped with a threaded Lucite cap. This prevents the inadvertent removal of the rubber stopper during the elution. The resin bed is supported by means of a layer of Teflon shavings.

The column is filled with 80 g of a strongly basic anion-exchange resin, 200 to 400 mesh, chlordie form, after the fine particles are removed from the resin by means of several decantations with water. This quantity of resin will give a column bed 20.3 cm long which is adequate for the separations in the procedure given below. A 2.22 cm polyethylene sphere inserted in the column serves to maintain a level surface in the resin bed. Without the sphere, the introduction of the sample solution and the eluents to the column disturbs the surface of the resin in such a manner as to cause the sample constituents to be eluted in diagonal instead of horizontal bands. The polyethylene sphere breaks the fall of the solutions sufficiently so as to keep the surface level and give horizontal bands during the elution. Polyethylene is an ideal material for this purpose since it rests on the resin bed when the column is empty, serves as a shield when the solutions are being added to the column, and floats to the surface during the elution so as to give the eluents free access to the entire cross section of the column. In addition, polyethylene is not wet by the solution and as a result the last quantities of the sample solution are easily rinsed on to the resin bed. The freshly prepared ion-exchange column should be washed several times alternately with 50 ml portions of 9 M HCl and 0.5 M HCl followed by 100 ml of 2.5% HF. A column which has been in use previously may be prepared for separations merely by washing with 50 ml of 2.5% HF.

2. *Column separations.* Take 10 ml of test specimen and evaporate to dryness on a steam bath. Take up the sample residue in 5 ml of concentrated HF plus 25 ml of water. Warm on the steam bath for about 10 minutes and then add an additional 75 ml of water. Transfer the sample solution to the anion-exchange column with 2.5% HF and after the sample solution is rinsed on to the resin bed, elute with 250 ml of 2.5% HF. This eluate will contain chromium; the column retains molybdenum. Elute the molybdenum with 300 ml of a solution which is 20% HF - 25% HCl.

⁽¹⁾See Reference 2.

3. To the molybdenum fraction add 6 ml of 1:1 sulfuric acid and evaporate the solution to fumes of sulfuric in order to remove the fluoride. Cool to room temperature and add several grams of ice followed by 25 ml of water, and 1:1 ammonium hydroxide until the molybdic acid just redissolves. Add 20 ml of the sodium acetate-acetic acid buffer and if necessary adjust the pH to approximately 4.8 with dilute acid. Add a small excess of 8-hydroxyquinoline in dilute acetic acid. Bring the solution to a boil on the hot plate and then digest for about 5 minutes at about 80 to 90°C. Cool the solution to room temperature and filter the molybdenum 8-hydroxyquinolate on a weighed Gooch crucible. Wash the precipitate several times with water, dry at 110°C for 1 hour and weigh the crucible plus precipitate. The factor for molybdenum is 0.2305.

c. Sulfates (Modified Kocour Method)

1. Allow specimen to cool to room temperature.
2. Add 5 ml of sulfate solution "A" to an aluminum-shielded glass centrifuge tube.
3. Pipette 20 ml of specimen into the tube.
4. Shake stoppered tube vigorously for 1 minute. Replace tube into shield and let stand for 5 minutes.
5. Centrifuge at 1000 rpm until all sediment has settled to the bottom of the tube; be sure to balance tubes in pairs.
6. Obtain "blank" reading by recording level in the tube.
7. Add 5 ml of sulfate solution "B" to the specimen.
8. Shake stoppered tube vigorously for 1 minute. Replace tube into shield and let stand for 5 minutes.
9. Centrifuge at 1000 rpm until all sediment has settled to the bottom of the tube.
10. Record level in the tube; subtract blank (Step 6) for corrected reading.

d. Sulfates (Optional Method)

1. Pipette 5 ml of specimen into 400 ml beaker and add 175 to 250 ml water.
2. Add 10 ml each of hydrochloric acid, formula 30 alcohol, and glacial acetic acid. Boil for 10 minutes.
3. Add 10 ml barium chloride solution and boil for 2 to 3 additional minutes. Remove from hot plate and let stand for 2 to 3 hours.
4. Using Number 42 Whatman paper, filter the specimen. Wash the paper and precipitate well.
5. Place paper and precipitate in weighed porcelain crucible.
6. Char and ignite at 538°C for 1 hour.
7. Cool and weigh.

INSPECTION PROCEDURE FOR ELECTRODEPOSITED CHROMIUM-MOLYBDENUM COATINGS

Procedures for testing the thickness, adhesion, hardness, and porosity of electrodeposited chromium-molybdenum coatings are detailed in Appendix C, Section 4 (Quality Assurance Provisions). Also included is a procedure to confirm compliance with hydrogen embrittlement relief requirements. Table I outlines the requirements to be met, the preparation of suitable test specimens, and the accompanying test procedures.

SECTION III

CONCLUSIONS

EVALUATION OF A NEW ETCHING TECHNIQUE

- The use of a hot sodium chromate-hydrofluoric acid etching solution (250 g/l $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$, 48 m/l concentrated HF, 82 to 100°C, immersion time = 20 min) increases the strength of the Cr-Mo/titanium substrate bond by at least 75% compared to the etch used previously (20 g/l $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$, 2.1 ml/l concentrated HF, room temperature, immersion time = 15 sec).

COATING OF AUGMENTOR NOZZLE SUPPORT BEARINGS FOR ENGINE TESTING

- In a 520-hr engine test, chromium-molybdenum coated titanium uniballs showed no failures, as opposed to a 30% failure rate for Bill-of-Material steel uniballs.

EVALUATION OF VARIOUS DRY-FILM LUBRICANTS

- Based on its wear and solvent resistance, PWA 474 (Dow Corning 3400A) is recommended for use with chromium-molybdenum on both titanium and steel alloy substrates.

SECTION IV

RECOMMENDATIONS

The following recommendations are made for continued development and evaluation of chromium-molybdenum coatings.

- Monitor the testing of engine P-652, the J52 engine with Cr-Mo plated first-stage compressor blades, and subject these blades to metallographic analysis after test completion.
- Continue to evaluate the coating's effectiveness as an antigallant on both titanium and steel uniballs, using test rigs and experimental engines.
- Evaluate chromium-molybdenum as a potential solution to other galling problems as they arise.

APPENDIX A
CHROMIUM-MOLYBDENUM
APPLICATION PROCEDURE FOR TITANIUM

SOLUTION COMPOSITION:

Self-Regulating Plating Bath

Unichrome ⁽¹⁾	300 g/l (40 oz/gal)
Ammonium Molybdate (NH ₄) ₆ Mo ₇ O ₂₄ · 4H ₂ O	75 g/l (10 oz/gal)

1. Dissolve the chromic acid in deionized water at room temperature.
2. Dissolve the ammonium molybdate in deionized water at 60 ± 5°C (140 ± 10°F).
3. Add the ammonium molybdate solution to the chromic acid and adjust the volume to operating level.

Vapor Blast

Novaculite 200 ⁽²⁾	480 to 720 g/l (4 to 6 lb/gal)
Antisolidifying Compound ⁽³⁾	113g/22.7 kg (4 oz/50 lb)
Corrosion Inhibitor ⁽⁴⁾	4 ml/l (15 ml/gal)

1. Fill tank to about 3/4 of operating level with water.
2. Add abrasive slowly while circulating pump is on.
3. Add antisolidifying compound.
4. Add corrosion inhibitor.
5. Fill to operating level.

Etch Solution

Hydrofluoric Acid (48%)	48 ml/l (180 ml/gal)
Sodium Chromate	250 g/l (33 oz/gal)

1. Dissolve the required amount of sodium chromate in deionized water.
2. Add required amount of hydrofluoric acid.
3. Raise temperature to 82 to 100°C (180 to 212°F).

⁽¹⁾M&T Chemicals, Inc., SRHS CR 110

⁽²⁾Vapor Blasting Manufacturing Co., NVB No. 200

⁽³⁾Vapor Blasting Manufacturing Co., No-Pak

⁽⁴⁾Reilly-Whiteman-Walton Co., X61-60 Water Conditioner.

PROCEDURE FOR PLATING:

1. Mask area not to be plated.
2. Vapor blast surface to be plated with wet abrasive at 0.4 to 0.7 MPa (50 to 100 psi).
3. Rinse thoroughly in clean running water. Cleaned surface must not be allowed to dry prior to plating. Submerge part in deionized water.
4. Etch for 20 min.
5. Rinse thoroughly in deionized water.
6. Using lead anodes, immerse part in plating solution with current "off."
7. Turn on power supply and raise current to 11.7 A/dm^2 (0.75 A/in.^2) for 2.5 min.
8. Plate at 46.7 A/dm^2 (3.0 A/in.^2) for 30 min with pulse plater set at desired "on"/"off" cycle. Setting off-time to zero results in direct current plating.
9. Rinse thoroughly in deionized water.
10. Heat-treat in vacuum as follows:

Ti 8Al-1Mo-1V:	954°C (1750°F) for 1 hr
followed by	593°C (1100°F) for 8 hr

Ti 6Al-4V:	760°C (1400°F) for 3 hr
followed by	538°C (1000°F) for 6 hr

For other titanium alloys, the diffusion heat-treat cycle should be compatible with previous heat-treat history and selected in accordance with MIL-H-81200.

11. (Option) — Vapor blast surfaces.

APPENDIX B

CHROMIUM-MOLYBDENUM APPLICATION PROCEDURE FOR STEELS

SOLUTION COMPOSITION:

Self-Regulating Plating Bath

Unichrome ⁽¹⁾	300 g/l (40 oz/gal)
Ammonium Molybdate (NH ₄) ₆ Mo ₇ O ₂₄ · 4H ₂ O	75 g/l (10 oz/gal)

1. Dissolve the chromic acid in deionized water at room temperature.
2. Dissolve the ammonium molybdate in deionized water at 60 ± 5°C (140 ± 10°F).
3. Add the ammonium molybdate solution to the chromic acid and adjust the volume to operating level.

STRESS RELIEF TREATMENT

All steel parts having an ultimate tensile strength of 1,034 MPa (150,000 psi) and above, which are machined, ground, cold-formed or cold-straightened, shall be baked at a minimum of 191 ± 14°C (375 ± 25°F) for 3 hr or more prior to cleaning and plating for the relief of damaging residual tensile stress.

PROCEDURE FOR PLATING:

1. Mask area not to be plated.
2. Anodic etch at 15.6 to 46.7 A/dm² (1 to 3 A/in.²) for 0.5 to 10 min. Etch in the following solution (preferred) or in the self-regulating plating bath described above:

Chromic Acid	225 to 300 g/l (30 to 40 oz/gal)
Temperature	32 to 54°C (90 to 130°F)

Note: Etching increases iron contamination in the plating bath; therefore, the use of this solution is recommended over the plating bath.

3. Using lead anodes, immerse part in plating solution with current "off."
4. Turn on power supply and raise current to 11.7 A/dm² (0.75 A/in.²) for 2.5 min.
5. Plate at 46.7 A/dm² (3.0 A/in.²) for 30 min with pulse plater set at desired "on"/"off" cycle. Setting off-time to zero results in direct current plating.
6. Rinse thoroughly in clear running water.

⁽¹⁾M&T Chemicals, Inc., SRHS CR 110

7. Embrittlement Relief

All coated steel parts having a hardness of Rockwell C40 and higher shall be baked at a minimum of $191 \pm 14^{\circ}\text{C}$ ($375 \pm 25^{\circ}\text{F}$) for 3 hr or more, within 4 hr after plating, to provide hydrogen-embrittlement relief.

8. (Option) — Vapor blast surfaces.

APPENDIX C

SUGGESTED MILITARY SPECIFICATION

CHROMIUM-MOLYBDENUM PLATING (ELECTRODEPOSITED)

1. SCOPE

1.1 Scope. This specification covers the requirements for electrodeposited chromium-molybdenum plating on steel and titanium alloys.

2. APPLICABLE DOCUMENTS

2.1 The following documents of the issue in effect on date of invitation for bids or request for proposal, form a part of this specification to the extent specified herein.

Military Specifications:

MIL-S-5002	Surface Treatments and Inorganic Coatings for Metal Surfaces of Weapons Systems
MIL-S-13165	Shot Peening of Ferrous Parts
MIL-H-81200	Heat Treatment of Titanium and Titanium Alloys
MIL-R-81841	Rotary Flap Peening of Metal Parts

Military Standards:

MIL-STD-105	Sampling Procedures and Tables for Inspection by Attributes
MIL-STD-1312	Fasteners, Test Methods

(Copies of Military Specifications and Standards required by contractors in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

Federal Specifications:

QQ-S-624	Steel Bar, Alloy, Hot Rolled and Cold Finished (General Purpose)
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(Activities outside the Federal Government may obtain copies of Federal Specifications, Standards and Handbooks as outlined under General Information in the Index of Federal Specifications and Standards and at the prices indicated in the Index. The Index, which includes cumulative monthly supplements as issued, is for sale on a subscription basis by the Superintendent of Documents, U. S. Government Printing Office, Washington, DC 20402.)

(Single copies of this specification and other Federal Specifications required by activities outside the Federal Government for bidding purposes are available without charge from Business Service Centers at the General Services Administration Regional Offices in Boston, New York, Washington, DC, Atlanta, Chicago, Kansas City, MO, Fort Worth, Denver, San Francisco, Los Angeles, and Seattle.)

(Federal Government activities may obtain copies of Federal Specifications, Standards, and Handbooks and the Index of Federal Specifications and Standards from established distribution points in their agencies.)

2.2 Other Publications. The following documents form a part of this specification to the extent specified herein. Unless a specific issue is identified, the issue in effect on date of invitation for bids or request for proposal shall apply.

American Society for Testing and Materials (ASTM) Standards:

ASTM B-481	Preparation of Titanium and Titanium Alloys for Electroplating
ASTM B-487	Measurement of Metal and Oxide Coating Thickness by Microscopic Examination of a Cross Section
ASTM B-499	Measurement of Coating Thicknesses by the Magnetic Method: Nonmagnetic Coatings on Magnetic Basis Metal
ASTM B-504	Measuring the Thickness of Metallic Coatings by the Coulometric Method
ASTM B-529	Measurement of Coating Thicknesses by the Eddy-Current Test Method: Nonconductive Coatings on Nonmagnetic Basis Metals
ASTM B-578	Measurement of Microhardness of Electroplated Coatings
ASTM E-8	Tension Testing of Metallic Materials

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.)

3. REQUIREMENTS

3.1 Materials and Operating Conditions. The materials used shall be such as to produce platings which meet the requirements of this specification. The deposition of chromium-molybdenum shall be out of a chromic acid type bath supplemented with a molybdate salt. Bath composition and operating conditions shall be such as to produce a satisfactory bond between the basis metal and the coating.

3.2 General Requirements.

3.2.1 High Tensile Steel Parts. Unless otherwise specified (see 6.2), steel parts having an ultimate tensile strength greater than 1655 MPa (240,000 psi) shall not be plated without specific approval of the procuring activity.

3.2.2 Stress Relief Treatment. All steel parts having an ultimate tensile strength of 1034 MPa (150,000 psi) and above, which are machined, ground, cold formed, or cold straightened, shall be baked at a minimum of $191 \pm 14^{\circ}\text{C}$ ($375 \pm 25^{\circ}\text{F}$) for three hours or more prior to cleaning and plating for the relief of damaging residual tensile stresses. When peening is required (see 3.3.3 and 3.3.5), thermal stress relief shall be performed prior to shot or rotary flap peening. Stress relief of titanium parts shall be in accordance with MIL-H-81200.

3.2.3 Surface Preparation.

3.2.3.1 Cleaning. Unless otherwise specified (see 6.2), all steel parts shall be cleaned in accordance with MIL-S-5002. Other base metals shall be cleaned by methods which shall not damage the substrate and shall not interfere with adhesion of the deposit. Vapor blasting is preferred for cleaning titanium parts.

3.2.3.2 Activating. Unless otherwise specified (see 6.2), all steel parts shall be activated by electrolytic etching in a chromic acid type bath. Titanium parts shall be activated in accordance with ASTM B-481, Process No. 1, omitting the pickling step, 2.2.1.2.

3.2.4 Plating Application. Unless otherwise specified (see 6.2), the plating shall be applied after all base metal heat treatments and mechanical operations, such as machining, brazing, welding, forming, and perforating of the article, have been completed.

3.2.5 Embrittlement Relief. All coated steel parts having a hardness of Rockwell C40 and higher shall be baked at a minimum of $191 \pm 14^{\circ}\text{C}$ ($375 \pm 25^{\circ}\text{F}$) for three hours or more, within four hours after plating, to provide hydrogen embrittlement relief (see 6.4). The baked parts, when tested in accordance with 4.5.5, shall not crack or fail by fracture (see 4.4.3.5). Plated springs and other parts subject to flexure shall not be flexed prior to hydrogen embrittlement relief treatment.

3.2.6 Diffusion Heat-Treat Cycle. All coated titanium parts should be heat treated to facilitate adhesion of the deposit and to provide hydrogen embrittlement relief. This diffusion cycle should be compatible with the previous heat-treat history of the base metal and selected in accordance with MIL-H-81200 unless otherwise specified (see 6.2). Titanium alloys not covered by MIL-H-81200 should be vacuum heat treated at 760°C (1400°F) for 3 hour.

3.2.7 Coverage. Unless otherwise specified (see 6.2), the plating shall cover all surfaces including roots of threads, corners, and recesses.

3.2.8 Boundaries. Boundaries of chromium-molybdenum plating which covers only a portion of the surface shall be free from beads, nodules, jagged edges, and other irregularities.

3.2.9 Finish. A gray matte finish, relatively smooth and free from blackened or frosty areas, shall be acceptable. Shading will vary depending on the molybdenum content of the deposit.

3.3 Processing of Steel Parts. Unless otherwise specified (see 6.2), steel parts shall be processed in accordance with the procedural instructions of the procuring activity as follows:

3.3.1 Class 1. Parts shall be plated or plated and processed to specific dimensions in accordance with procedures and criteria specified by the procuring activity. Parts not covered by procedural instructions which do not specify baking procedures shall be baked in accordance with 3.2.5 after plating.

3.3.2 Class 2. Plated parts below Rockwell C40 hardness, which are subjected to static loads or designed for limited life under dynamic loads or combinations thereof, need not be peened prior to plating.

3.3.3 Class 3. Plated parts below Rockwell C40 hardness, which are designed for unlimited life under dynamic loads, shall be peened in accordance with MIL-S-13165 or MIL-R-81841 prior to plating. Unless otherwise specified in the applicable drawings, the peening shall be accomplished on all surfaces for which the plating is required and on all immediately adjacent surfaces when they contain notches, fillets, or other abrupt changes in section size where stress will be concentrated.

3.3.4 Class 4. Plated parts, Rockwell C40 hardness or above, which are subjected to static loads or designed for limited life under dynamic loads or combinations thereof, shall be baked in accordance with 3.2.5 after plating. The load for the static load test (see 4.5.5, the embrittlement relief test) shall be as specified in the contract, order, or applicable drawing (see 6.2).

3.3.5 Class 5. Plated parts, Rockwell C40 hardness or above, which are designed for unlimited life under dynamic loads, shall be peened in accordance with MIL-S-13165 or MIL-R-81841 prior to plating. Unless otherwise specified in the applicable drawings, the peening shall be accomplished on all surfaces for which the plating is required and on all immediately adjacent surfaces when they contain notches, fillets, or other abrupt changes in section size where stress will be concentrated. The plated parts shall be baked in accordance with 3.2.5 after plating. The load for the static load test (see 4.5.5, the embrittlement relief test) and the dynamic load conditions shall be as specified in the contract, order, or applicable drawing (see 6.2).

3.4 Detail Requirements.

3.4.1 Thickness. The minimum, maximum, or range of thickness for chromium-molybdenum plating shall be as specified in the contract, purchase order, or on the applicable drawing (see 6.2). If a thickness is not specified, the minimum thickness for the finished part shall be 0.0005 in., or 0.5 mil (0.013 mm). The thickness requirement shall apply after all metal finishing and post-plating grinding operations have been completed.

3.4.2 Adhesion. The adhesion of the plating and any underplate shall be such that when examined at a magnification of approximately 4 diameters, neither the plating, nor any electrodeposited underplate, shall show separation from the base metal or from each other at their common interface(s) when subjected to the test described in 4.5.2. The interface between a plating and the base metal is the surface of the base metal before plating. The formation of cracks in the base metal or the plate which do not result in flaking, peeling, or blistering of the plate shall not be cause for rejection.

3.4.3 Hardness. The minimum hardness of a cross section of the plating, when subjected to the microhardness test detailed in 4.5.3, shall be 200 Vickers Hardness Number (VHN).

3.4.4 Porosity. The chromium-molybdenum plating, by being as free from porosity as possible, shall be capable of protecting steel parts from corrosion due to pits, pores, or cracking. When subjected to the test detailed in 4.5.4, specimens shall show no more than a total of 15 isolated spots or pits, none larger than 0.79 mm (0.03 in.) in diameter, in a total of 967.8 sq cm (150 sq in.) of test area grouped from five or more test pieces; nor more than five isolated spots or pits, none larger than 0.79 mm (0.03 in.) in a total of 193.6 sq cm (30 sq in.) from one or more test pieces, except those areas within 1.59 mm (0.06 in.) from identification markings and contact marks after processing.

3.5 Workmanship.

3.5.1 Base Metal. The base metal shall be free from visible defects that will be detrimental to the appearance or protective value of the plating. The base metal shall be subjected to such cleaning and plating procedures as necessary to yield deposits herein specified.

3.5.2 Plating. The plating shall be smooth, fine grained, adherent, uniform in appearance, free from blisters, pits, nodules, excessive edge build-up, and other defects. The plating shall show no indication of contamination or improper operation of equipment used to produce the deposit, such as excessively powdered or darkened plating, build-up, and other defects. The size and number of contact marks shall be at a minimum consistent with good practice. The location of contact marks shall be in areas of minimum exposure to service environmental conditions where important to the function of the part. Superficial staining which has been demonstrated as resulting from rinsing or slight discoloration resulting from baking operations to relieve embrittlement, as specified above (see 3.2.5), shall not be cause for rejection. All details of workmanship shall conform to the best practice for high quality plating.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for Inspection. Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract or order, the supplier may use his own or any other facilities herein, unless disapproved by the Government. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure that supplies and services conform to prescribed requirements.

4.2 Classification of Inspection. The inspection requirements specified herein are classified as follows:

- a. Production Control Inspection (see 4.3)
- b. Quality Conformance Inspection (see 4.4).

4.3 Production Control Inspection.

4.3.1 Control Records. When specified in the contract or order (see 6.2), the supplier shall maintain a record of each processing bath, showing all additional chemicals or treatment solutions to the unit, the results of all chemical analyses performed, and the quantity of parts plated during operation. Upon request of the procuring activity, such records, as well as reports of the test results, shall be made available. These records shall be maintained for not less than one year after completion of the contract or purchase order.

4.3.2 Production Control. The equipment, procedures, and operations employed by a supplier shall be capable of producing high quality electrodeposited platings as specified in this document. When specified by the procuring activity (see 6.2), the supplier, prior to production, shall demonstrate the capability of the process used to show freedom from hydrogen embrittlement damage as indicated by satisfactory behavior of specimens prepared (see 6.2.2) and tested in accordance with 4.3.2.1 to comply to the requirements of MIL-S-5002 for preproduction process qualifications.

4.3.2.1 Preproduction Control. For preproduction control, four round notched steel specimens shall be prepared in accordance with 4.4.4.3 from four individual heats for a total of 16 specimens, using the specified steel alloy for which preproduction examinations of the process is to be demonstrated. Specimens shall be heat treated to the maximum tensile strength representing production usage. The specimens shall be given the same pretreatments and treatments proposed for production. The specimens shall be subject to the test detailed in 4.5.5. The process shall be considered satisfactory if all specimens show no indication of cracks or failure. The test results and production control information shall be submitted to the procuring activity for approval. Until approval has been received parts shall not be plated.

4.3.3 Frequency of Tests. To assure continuous control of the process as required by MIL-S-5002 and to prevent detrimental hydrogen embrittlement during production, the satisfactory behavior of specimens prepared and tested in accordance with Table I shall be made once each month or more frequently if required by the procuring activity. The results of tests made to determine conformation of electrodeposited platings to all requirements of this specification are acceptable as evidence of the properties being obtained with the equipment and procedures employed.

4.3.4 Production Control Specimens. Test specimens for production control shall be prepared in accordance with 4.4.4, 4.4.4.1 and 4.4.4.2, as applicable for the thickness, adhesion, hardness, and porosity tests detailed in Table I. Specimens for the production control embrittlement relief test shall be four round notched steel specimens of alloy steel 4340, conforming to QQ-S-624, heat treated to the maximum tensile strength from one or more heats, and prepared in accordance with 4.4.4.3.

TABLE I. PRODUCTION CONTROL TESTS AND SPECIMENS

<i>Test</i>	<i>Requirement Paragraphs</i>	<i>Specimen Preparation Paragraph⁽¹⁾</i>	<i>Test Reference Paragraphs</i>
Thickness	3.4.1	4.4.4 and 4.4.4.1	4.5.1
Adhesion	3.4.2	4.4.4 and 4.4.4.1	4.5.2
Hardness	3.4.3	4.4.4 and 4.4.4.1	4.5.3
Porosity	3.4.4	4.4.4 and 4.4.4.2	4.5.4
Hydrogen Embrittlement Relief	3.2.5	4.4.4 and 4.4.4.3	4.5.5

⁽¹⁾Standard alloy steels shall be used for production control specimens. The selection shall be at the option of the supplier; however, alloy steels such as AISI or SAE numbers 4130, 4135, 4140, 4145, 4340, 8645 and 8740 conforming to QQ-S-624 shall be used.

4.4 Quality Conformance Inspection.

4.4.1 Lot. A lot shall consist of plated articles of the same base metal composition, class, deposition form and finish, plated and treated under the same conditions, and approximately the same size and shape submitted for inspection at one time.

4.4.2 Sampling for Visual Examination and Nondestructive Tests. Sampling for visual examination and nondestructive tests shall be conducted as directed by the procuring activity (see 6.2) in accordance with MIL-STD-105 or using Table II. A sample of coated parts or articles shall be drawn by taking at random from each lot the number of articles in accordance with MIL-STD-105, Level II, Acceptable Quality Level (AQL) 1.5 percent defective or as indicated in Table II. The lot shall be accepted or rejected according to the procedures in 4.4.2.1 for visual examination and 4.4.2.2 for plating thickness (nondestructive tests).

4.4.2.1 Visual Examination. Samples selected in accordance with 4.4.2 shall be examined for compliance with the requirements of 3.2.7, 3.2.8, 3.2.9, and 3.5.2 after plating. If the number of nonconforming articles exceeds the acceptance number for the sample, the lot represented by the sample shall be rejected.

4.4.2.2 Thickness of Plating (Nondestructive Tests). Samples selected in accordance with 4.4.2 shall be inspected and the plating thickness measured by the applicable test detailed in 4.5.1 at several locations on each article as defined in 3.4.1 for compliance with the requirement. Measurements on fastener hardware shall be made on locations defined in MIL-STD-1312, Test 12. The part or article shall be considered nonconforming if one or more measurements fail to meet the specified minimum thickness. If the number of defective items in any sample exceeds the acceptance number for the specified sample, the lot represented by the sample shall be rejected. Separate specimens (see 4.4.4.1) shall not be used for thickness measurements unless a need has been demonstrated.

TABLE II. SAMPLING FOR VISUAL EXAMINATION
AND NONDESTRUCTIVE TESTS

<i>Number of Items in Lot Inspections</i>	<i>Number of Items in Samples (Randomly Selected)</i>	<i>Acceptance Number (maximum number of sample items nonconforming to any test)</i>
15 or less	7 ¹	0
16 to 40	10	0
41 to 110	15	0
111 to 300	25	1
301 to 500	35	1
501 and over	50	2

¹If the number of items in the inspection lot is less than 7, the number of items in the sample shall equal the number of items in the inspection lot.

4.4.3 Sampling for Destructive Tests. A random sample of five plated parts or articles shall be taken from each lot for each destructive test or separately plated specimens shall be prepared in accordance with 4.4.4, 4.4.4.1, 4.4.4.2, and 4.4.4.3 to represent each lot. If the number of articles in the lot is five or less, the number of articles in the sample shall be specified by the procuring activity (see 6.2).

4.4.3.1 Thickness of Plating (Destructive Tests). If sampling and testing for the thickness of plating by nondestructive testing is not the option of the supplier, samples selected in accordance with 4.4.3 shall be measured for plating thickness by the applicable tests detailed in 4.5.1 at several locations as defined in 3.4.1 for compliance with the requirements. Measurements for fastener hardware shall be made at locations defined in MIL-STD-1312, Test 12. If the plating thickness at any place on any article or specimen is less than the specified minimum thickness, the lot shall be rejected. Separate specimens (see 4.4.4.1) shall not be used for thickness measurements unless a need has been demonstrated.

4.4.3.2 Adhesion (Destructive Tests). The articles or specimens used for the destructive thickness test (see 4.4.3.1), if of suitable size and form, may be used as the test pieces for the adhesion test to determine compliance with the requirements of 3.4.2. Failure of one or more of the test pieces shall constitute failure of the lot.

4.4.3.3 Hardness (Destructive Tests). When specified in the contract or offer (see 6.2), compliance with the requirements for hardness shall be determined. The articles or specimen used for the destructive thickness test (see 4.4.3.1), if of suitable size and form, may be used as the test pieces for examination to determine compliance with the requirement of 3.4.3. Failure of one or more of the test pieces shall constitute failure of the lot.

4.4.3.4 Porosity (Destructive Tests). When specified in the contract or order (see 6.2), compliance with the requirements for porosity of chromium-molybdenum on steel shall be determined. A set of five separate test specimens prepared in accordance with 4.4.4 and 4.4.4.2 in lieu of treated plated articles shall be used to determine compliance with the requirements for porosity (see 3.4.4). Failure of one or more of the test specimens shall reject the lot.

4.4.3.5 Hydrogen Embrittlement Relief (Destructive Tests). Unless otherwise specified in the contract or offer (see 6.2), conformance to the requirements of 3.2.5 for hydrogen embrittlement relief of treated steel parts shall be determined for those parts, comprising a lot, having a tensile strength of or heat treated to a tensile strength level of 1655 MPa (240,000 psi) or above and which will be subjected to a sustained tensile load in use. A random sample of five plated articles shall be taken from each lot or five specimens, prepared in accordance with 4.4.4 and 4.4.4.3, shall be used to represent the lot. When tested as specified in 4.5.5, cracks or failure by fracture shall be cause for rejection. Failure of one or more of the test pieces shall reject the lot.

4.4.4 Quality Conformance Specimen Preparation. When the plated articles are of such form, shape, size, and value as to prohibit use thereof, or are not readily adaptable to a test specified herein, or when destructive tests of small lot sizes are required, the test shall be made by the use of separate specimens plated concurrently with the articles represented. The separate specimens shall be of a base metal equivalent to that of the article represented. "Equivalent" base metal includes chemical composition, grade, condition and finish of surface prior to plating. For example, a cold-rolled steel surface should not be used to represent a hot-rolled steel surface. Due to the impracticality of forging or casting separate test specimens, hot-rolled specimens may be used to represent forged and cast-steel articles. The separate specimens may also be cut from the scrap casting when ferrous alloy castings are being plated. These separate specimens may be introduced into a lot at regular intervals prior to the cleaning operations, prior to plating, and shall not be separated therefrom until after completion of plating. Conditions affecting the plating of specimens, including the spacing, plating media, bath agitation, temperature, etc., in respect to other objects being plated shall correspond as nearly as possible to those affecting the significant surfaces of the articles represented. Separate specimens shall not be used for thickness measurements, however, unless the necessity for their use has been demonstrated.

4.4.4.1 Specimens for Thickness, Adhesion, and Hardness Tests. If separate specimens for thickness, adhesion, and hardness tests are required, they shall be strips approximately 25 mm (1 in.) wide, 102 mm (4 in.) long and 1 mm (0.04 in.) thick.

4.4.4.2 Specimens for Porosity Tests. If separate specimens for porosity tests are required, they shall be panels not less than 254 mm (10 in.) in length, 76 mm (3 in.) in width and approximately 1 mm (0.04 in.) thick.

4.4.4.3 Specimens for Embrittlement Relief. Separate specimens for embrittlement relief test shall be round notched specimens with the axis of the specimen (load direction) perpendicular to the short traverse grain flow direction. The configuration shall be in accordance with Figure 8 of ASTM E-8 for rounded specimens. Specimens shall have a 60 deg V-notch located approximately at the center of the gage length. The cross-section area at the root of the vee shall be approximately equal to half the area of the full cross-section area of the specimen's reduced section. The vee shall have a 0.254 ± 0.0127 mm (0.010 ± 0.0005 in.) radius of curvature at the base of the notch (see 6.2.2).

4.5 Tests.

4.5.1 Thickness. For nondestructive measurement of plating thickness on steel, procedures in accordance with ASTM B-499 (Magnetic Test Method) may be used. On titanium alloys, plating thickness may be measured with an eddy current-type gage per ASTM B-529. For destructive measurement of plating thickness, procedures in accordance with ASTM B-487 (Microscopic) or ASTM B-504 (Coulometric) may be used. In addition to the above, other procedures embodied in MIL-STD-1312, Test 12, may be used for thickness measurement of plated fastener hardware.

4.5.2 Adhesion. Adhesion may be determined by scooping the surface or shearing with a sharp edge, knife, or razor through the plating down to the base metal and examining at four diameters magnification for evidence of non-adhesion. Alternately, the article or specimen may be clamped in a vise and the projecting portion bent back and forth until rupture occurs. If the edge of the ruptured plating can be peeled back or if separation between the plating and the base metal can be seen at the point of rupture when examined at four diameters magnification, adhesion is not satisfactory.

4.5.3 Hardness. The hardness of plating shall be determined by a microhardness traverse in accordance with ASTM B-578, except that a Vicker indenter and 100 gram load shall be used. A minimum of three hardness readings shall be made to establish the base metal hardness in an area at least 3.175 mm (0.125 in.) from the outer surface or at mid radius of the cross section, whichever is less. Readings shall be taken at 0.013 mm (0.0005 in.) intervals starting at 0.025 mm (0.001 in.) from the outer surface in a staggered pattern until the pre-established base metal hardness is reached. The hardness reading may be plotted versus distance from the outer surface. The point at which the hardness shows a vast decrease may be taken as the limits of chromium-molybdenum plating.

4.5.4 Porosity. Prior to determining porosity of chromium-molybdenum on steel by the ferroxyl test, the specimen surface shall be cleaned to remove any oil or grease. Contamination removal shall be accomplished with any acceptable solvent in accordance with MIL-S-5002. A sheet of filter paper, saturated by dipping in a ferroxyl solution heated to 82 to 94°C (180 to 200°F), shall be applied to the flat surface of the specimen or of the article. The solution composition shall be as follows:

Potassium ferricyanide ($K_3Fe(CN)_6$)	1 gm
Sodium chloride (NaCl)	10 gm
Agar	10 gm
Water (distilled or deionized) to make 1 litre	

After 10 min, the heated filter paper shall be removed. Both the plated surface and the filter paper shall be examined. Where corrosion of the base metal will occur at pores or other defects due to the plating, dark blue spots will have been developed. Contact may further be assured by the use of a soft bristle brush moistened with the reagent solution. For a permanent record, the filter paper can be dried.

4.5.5 Embrittlement Relief. Compliance with 3.2.5 shall be determined with samples of plated parts as specified in 4.4.3.5. Parts such as spring pins, lock rings, etc., which are installed in holes or rods, shall be similarly assembled using the applicable parts specifications or drawings tolerances which impose the maximum sustained tensile load on the plated part. The selected samples shall be subjected to a sustained tensile load equal to 115 percent of the maximum design yield load for which the part was designed. Parts which require special fixtures, extreme loads to comply with the above requirements, or where the maximum design yield load is not known, may be represented by separate specimens prepared in accordance with 4.4.4.3. The notched samples shall be subjected to a sustained tensile load equal to 75 percent of the ultimate notch tensile strength of the material. The articles, parts or specimens shall be held under load for at least 200 hours and then examined for cracks or fracture.

5. PREPARATION FOR DELIVERY

5.1 Packaging and Packing. Preservation, packaging, and packing methods for electrodeposited plated parts or articles employed by a supplier shall be such as to preclude damaging during shipment and handling.

6. NOTES

6.1 Intended Use. Chromium-molybdenum plating is used for wear resistance, abrasion resistance, and such incidental corrosion protection of parts as the specified thickness of the plating may afford. Heavy deposits of the plating may be used for buildup of worn or undersized parts or for salvage purposes, and to provide protection against corrosive chemical environments.

6.2 Ordering Data. Purchasers should select the preferred options permitted herein and include the following information in procurement documents.

- a. Title, number, and date of this specification
- b. Class of plating (see 1.2, 3.3, 3.3.1, 3.3.2, 3.3.3, 3.3.4, and 3.3.5)
- c. When plating is to be applied, if other than specified (see 3.2.1, 3.2.4, 3.3, 3.3.1, 3.3.2, 3.3.3, 3.3.4, and 3.3.5)
- d. Surface preparation, if other than specified (see 3.2.3)
- e. Heat-treat cycle, if other than specified (see 3.2.6)
- f. Coverage, if other than specified (see 3.2.7)
- g. Surface finish, if particular finish required (see 3.2.9)
- h. Thickness of plating, as specified (see 3.3, 3.3.1, and 3.4.1)
- i. Control record requirement (see 4.3.1)
- j. Preproduction control examination (see 4.3.2)
- k. Sampling plan (see 4.4.2)
- l. Number of samples for destructive testing (see 4.4.3)
- m. Hardness, porosity, and hydrogen embrittlement tests, whether required for quality conformance inspection (see 4.4.3.3, 4.4.3.4, and 4.4.3.5).

6.2.1 The manufacturer of the base metal parts should provide the plating facility with the following data:

- a. Hardness of steel parts (see 3.2.1, 3.2.2, 3.2.5, and 3.3)
- b. Heat treatment for stress relief, whether has been performed or is required (see 3.2.2)
- c. Tensile loads required for embrittlement relief test, if applicable (see 3.2.5 and 4.5.5).

6.2.2 The manufacturer of the base metal parts should provide the plating facility with notched specimens (see 4.4.4.3) to be plated for conformance with 3.2.5 required for production control (see 4.3.2.1 and 4.3.4) and for lot acceptance (see 4.4.3 and 4.4.3.5).

6.3 Stress Relief. There is a hazard that hardened and tempered cold-worked or cold-straightened steel parts may crack during cleaning and plating. Such parts shall have a suitable heat treatment for stress relief prior to cleaning and plating (see 3.2.2).

6.4 Baking Time. For high strength steels (Rockwell C40 and above), it may be beneficial to extend the baking time to 23 hours to ensure complete hydrogen embrittlement relief (see 3.2.5).

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